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# 3,6-Bis(3,4,5-trimethoxyphenyl)-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazole

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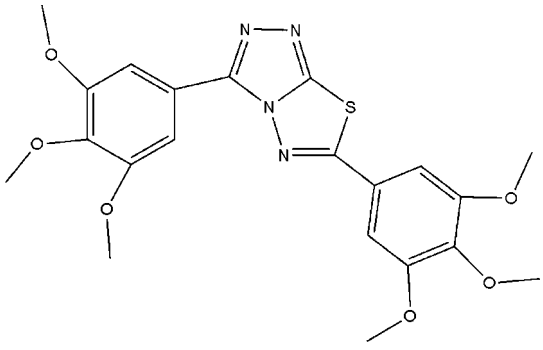
Key indicators: single-crystal X-ray study; $T$ = 113 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.044; $wR$ factor = 0.138; data-to-parameter ratio = 12.6.

In the molecule of the title compound, $C_{21}H_{22}N_4O_6S$, the planar central heterocyclic ring system is oriented with respect to the trimethoxyphenyl rings at dihedral angles of 2.60 (5) and 3.60 (6)°. Intramolecular C—H···N and C—H···S hydrogen bonds result in the formation of planar five- and six-membered rings. In the crystal structure, intermolecular C—H···O hydrogen bonds link the molecules. There is a C—H···$\pi$ contact between a methyl group and a trimethoxyphenyl ring, and a $\pi$–$\pi$ contact between the central heterocyclic ring system and a trimethoxyphenyl ring [centroid–centroid distance = 3.640 (1) Å].

## Related literature

For general background, see: Karabasanagouda *et al.* (2007); Mathew *et al.* (2007).



## Experimental

### Crystal data

$C_{21}H_{22}N_4O_6S$
$M_r$ = 458.49
Triclinic, $P\overline{1}$
$a$ = 8.6762 (17) Å
$b$ = 8.9289 (18) Å
$c$ = 13.735 (3) Å
$\alpha$ = 94.50 (3)°
$\beta$ = 90.82 (3)°
$\gamma$ = 90.47 (3)°
$V$ = 1060.6 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.20 mm$^{-1}$
$T$ = 113 (2) K
0.22 × 0.20 × 0.10 mm

### Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min}$ = 0.957, $T_{max}$ = 0.980
6899 measured reflections
3720 independent reflections
3102 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.022

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.137
$S$ = 1.19
3720 reflections
295 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.93 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.56 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2···N4 | 0.93 | 2.36 | 3.047 (3) | 130 |
| C14—H14···S1 | 0.93 | 2.72 | 3.130 (3) | 108 |
| C19—H19$B$···O3$^{i}$ | 0.96 | 2.53 | 3.384 (2) | 148 |
| C21—H21$B$···O1$^{ii}$ | 0.96 | 2.43 | 3.331 (3) | 156 |
| C19—H19$C$···$Cg3^{iii}$ | 0.96 | 3.30 | 4.057 (3) | 137 |

Symmetry codes: (i) $x+1, y, z-1$; (ii) $-x, -y, -z+2$; (iii) $-x+1, -y+1, -z$. $Cg3$ is the centroid of the trimethoxyphenyl ring C1–C6.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2498).

## References

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Mathew, V., Keshavayya, J., Vaidya, V. P. & Giles, D. (2007). *Eur. J. Med. Chem.* **42**, 823–840.
Rigaku/MSC. (2005). *CrystalClear* and *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2008). E**64**, o1577 [doi:10.1107/S1600536808022502]

## 3,6-Bis(3,4,5-trimethoxyphenyl)-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazole

**Hai-Tang Du, Hai-Jun Du and Weiyi Zhou**

### S1. Comment

1,2,4-Triazole and 1,3,4-thiadiazole represent one of the most biologically active classes of compounds, possessing a wide spectrum of activities. Various substituted 1,2,4-triazolo[3,4-b]-1,3,4-thiadiazoles are associated with diverse pharmacological activities such as antimicrobial (Karabasanagouda *et al.*, 2007) and anti-inflammatory activity (Mathew *et al.*, 2007). We report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig. 1) the bond lengths and angles are within normal ranges. Rings A (C1-C6), B (N1-N3/C10/C11), C (S1/N3/N4/C11/C12) and D (C13-C18) are, of course, planar, and the dihedral angles between them are A/B = 3.42 (6)°, A/C = 1.96 (5)°, A/D = 4.76 (5)°, B/C = 1.65 (6)°, B/D = 3.91 (6)° and C/D = 3.42 (5)°. So, the rings are nearly coplanar. The intramolecular C-H···N and C-H···S hydrogen bonds (Table 1) result in the formation of planar six- and five-membered rings E: (N3/N4/C1/C2/C10/H2) and F (S1/C12-C14/H14), in which they are oriented with respect to the planar central heterocylic ring system at dihedral angles of 1.56 (5)° and 4.00 (5)°, respectively.

In the crystal structure, intermolecular C-H···O hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure. A C—H···$\pi$ contact (Table 1) between the trimethoxyphenyl ring and the methyl group and a $\pi$—$\pi$ contact between B and D rings Cg2···Cg4$^{i}$ [symmetry code: (i) 1 - x, 1 - y, -z, where Cg2 and Cg4 are centroids of the rings B (N1-N3/C10/C11) and D (C13-C18), respectively] further stabilize the structure, with centroid-centroid distance of 3.640 (1) Å.

### S2. Experimental

For the preparation of the title compound, 4-amino-5-(3,4,5-trimethoxyphenyl) -4H-1,2,4-triazole-3-thiol (0.01 M) and 3,4,5-trimethoxybenzoic acid (0.01 M) were dissolved in dry phosphorous oxychloride (10 ml). The resulted solution was further heated under reflux for 7 h. The reaction mixture was cooled to room temperature and the mixture was gradually poured onto crushed ice with stirring. Finally, powdered potassium carbonate and the required amount of solid potassium hydroxide were added until the pH of the mixture was raised to 8, to remove the excess of phosphorous oxychloride. The mixture was allowed to stand overnight and the solid was separated. It was filtered, washed with cold water, and then dried. Crystals suitable for X-ray analysis were obtained by the recrystallization of the solid residue from a mixture of N,N-dimethyl- formamide/ethanol (1:1) by slow evaporation at room temperature.

### S3. Refinement

H atoms were positioned geometrically, with C-H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = xU_{eq}(C)$, where x = 1.5 for methyl H and x = 1.2 for aromatic H atoms.

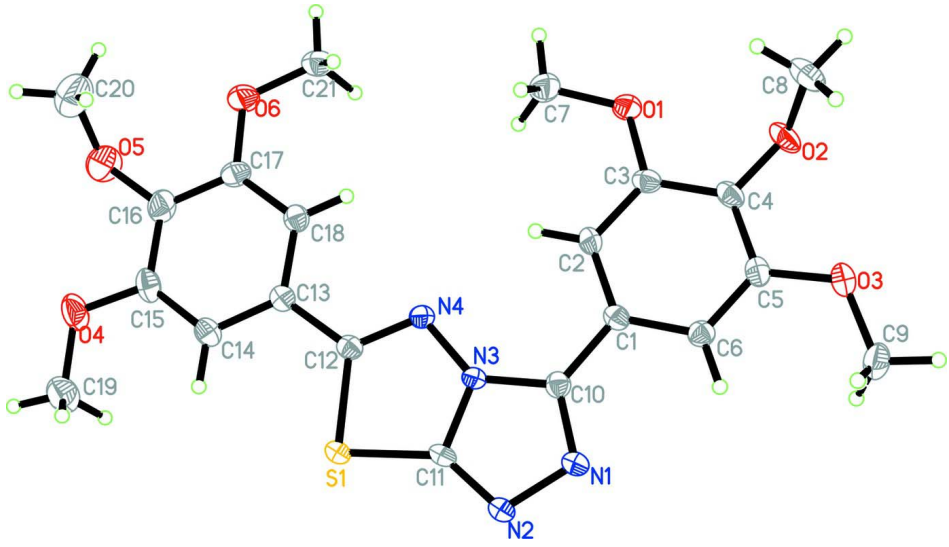


**Figure 1**

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

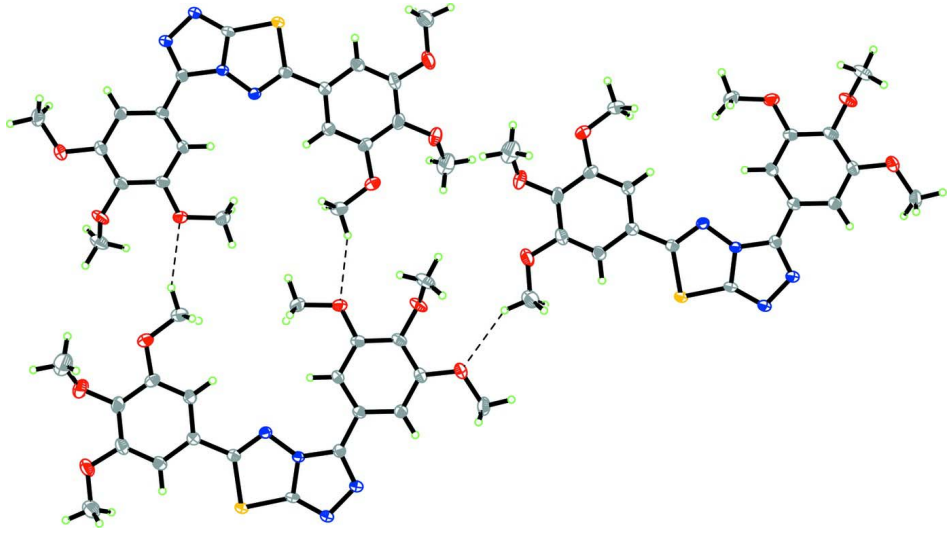

**Figure 2**

A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

### 3,6-Bis(3,4,5-trimethoxyphenyl)-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazole

*Crystal data*

$C_{21}H_{22}N_4O_6S$
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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.6762\ (17)$ Å
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$\alpha = 94.50\ (3)°$
$\beta = 90.82\ (3)°$
$\gamma = 90.47\ (3)°$
$V = 1060.6\ (4)$ Å$^3$
$Z = 2$
$F(000) = 480$
$D_x = 1.436$ Mg m$^{-3}$
Melting point: 423 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3022 reflections
$\theta = 2.8–27.9°$

$\mu$ = 0.20 mm$^{-1}$
$T$ = 113 K

Prism, colorless
0.22 × 0.20 × 0.10 mm

*Data collection*

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Confocal monochromator
$\omega$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min}$ = 0.957, $T_{max}$ = 0.980

6899 measured reflections
3720 independent reflections
3102 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.022
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.8°
$h$ = −7→10
$k$ = −10→10
$l$ = −15→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.137
$S$ = 1.19
3720 reflections
295 parameters
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0797P)^2 + 0.2575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.93 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.56 e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2$sigma($F^2$) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.50010 (6) | 0.76590 (6) | 0.89162 (4) | 0.01987 (19) |
| O1 | −0.04182 (18) | 0.27249 (17) | 1.14986 (12) | 0.0270 (4) |
| O2 | −0.17317 (18) | 0.40003 (18) | 1.31069 (12) | 0.0282 (4) |
| O3 | −0.09095 (19) | 0.67584 (18) | 1.38496 (11) | 0.0290 (4) |
| O4 | 0.6387 (2) | 0.3996 (2) | 0.57836 (12) | 0.0352 (4) |
| O5 | 0.5139 (3) | 0.1310 (2) | 0.59409 (12) | 0.0457 (5) |
| O6 | 0.3352 (2) | 0.07371 (18) | 0.74567 (12) | 0.0305 (4) |
| N1 | 0.2808 (2) | 0.88200 (19) | 1.13007 (13) | 0.0207 (4) |
| N2 | 0.3818 (2) | 0.9307 (2) | 1.06090 (13) | 0.0220 (4) |
| N3 | 0.31915 (19) | 0.69318 (18) | 1.02331 (12) | 0.0161 (4) |
| N4 | 0.3240 (2) | 0.56265 (19) | 0.96337 (12) | 0.0173 (4) |
| C1 | 0.1406 (2) | 0.6468 (2) | 1.16140 (15) | 0.0175 (4) |
| C2 | 0.1044 (2) | 0.5002 (2) | 1.12685 (15) | 0.0197 (5) |
| H2 | 0.1477 | 0.4583 | 1.0695 | 0.024* |

| | | | | |
|---|---|---|---|---|
| C3 | 0.0031 (2) | 0.4170 (2) | 1.17875 (16) | 0.0204 (5) |
| C4 | −0.0631 (2) | 0.4788 (2) | 1.26440 (16) | 0.0209 (5) |
| C5 | −0.0226 (2) | 0.6260 (2) | 1.29933 (15) | 0.0205 (5) |
| C6 | 0.0775 (2) | 0.7102 (2) | 1.24824 (14) | 0.0193 (5) |
| H6 | 0.1029 | 0.8082 | 1.2713 | 0.023* |
| C7 | 0.0400 (3) | 0.1984 (3) | 1.07097 (18) | 0.0311 (6) |
| H7A | 0.0241 | 0.2505 | 1.0131 | 0.047* |
| H7B | 0.0029 | 0.0969 | 1.0596 | 0.047* |
| H7C | 0.1480 | 0.1981 | 1.0871 | 0.047* |
| C8 | −0.1128 (3) | 0.3006 (3) | 1.37736 (19) | 0.0356 (6) |
| H8A | −0.0456 | 0.2300 | 1.3435 | 0.053* |
| H8B | −0.1961 | 0.2475 | 1.4051 | 0.053* |
| H8C | −0.0561 | 0.3572 | 1.4285 | 0.053* |
| C9 | −0.0618 (3) | 0.8283 (3) | 1.41985 (17) | 0.0354 (6) |
| H9A | 0.0469 | 0.8435 | 1.4308 | 0.053* |
| H9B | −0.1152 | 0.8504 | 1.4800 | 0.053* |
| H9C | −0.0976 | 0.8936 | 1.3722 | 0.053* |
| C10 | 0.2438 (2) | 0.7397 (2) | 1.10791 (15) | 0.0184 (5) |
| C11 | 0.4024 (2) | 0.8140 (2) | 0.99828 (15) | 0.0178 (4) |
| C12 | 0.4137 (2) | 0.5860 (2) | 0.89114 (15) | 0.0175 (4) |
| C13 | 0.4421 (2) | 0.4679 (2) | 0.81339 (15) | 0.0179 (5) |
| C14 | 0.5310 (2) | 0.4968 (3) | 0.73302 (15) | 0.0218 (5) |
| H14 | 0.5742 | 0.5916 | 0.7283 | 0.026* |
| C15 | 0.5545 (3) | 0.3832 (3) | 0.66039 (16) | 0.0260 (5) |
| C16 | 0.4910 (3) | 0.2408 (3) | 0.66800 (16) | 0.0285 (5) |
| C17 | 0.3990 (3) | 0.2149 (2) | 0.74754 (16) | 0.0233 (5) |
| C18 | 0.3746 (2) | 0.3271 (2) | 0.82064 (15) | 0.0201 (5) |
| H18 | 0.3140 | 0.3090 | 0.8739 | 0.024* |
| C19 | 0.6870 (3) | 0.5477 (3) | 0.56178 (19) | 0.0390 (6) |
| H19A | 0.5995 | 0.6131 | 0.5647 | 0.058* |
| H19B | 0.7325 | 0.5476 | 0.4985 | 0.058* |
| H19C | 0.7615 | 0.5827 | 0.6109 | 0.058* |
| C20 | 0.5942 (4) | −0.0017 (3) | 0.6225 (2) | 0.0512 (8) |
| H20A | 0.6491 | 0.0223 | 0.6830 | 0.077* |
| H20B | 0.6657 | −0.0343 | 0.5727 | 0.077* |
| H20C | 0.5207 | −0.0806 | 0.6307 | 0.077* |
| C21 | 0.2401 (3) | 0.0431 (3) | 0.82573 (17) | 0.0280 (5) |
| H21A | 0.2991 | 0.0570 | 0.8855 | 0.042* |
| H21B | 0.2029 | −0.0587 | 0.8171 | 0.042* |
| H21C | 0.1543 | 0.1105 | 0.8286 | 0.042* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0218 (3) | 0.0164 (3) | 0.0217 (3) | −0.0034 (2) | 0.0056 (2) | 0.0029 (2) |
| O1 | 0.0264 (9) | 0.0190 (8) | 0.0353 (9) | −0.0065 (7) | 0.0089 (7) | −0.0005 (7) |
| O2 | 0.0202 (8) | 0.0324 (9) | 0.0340 (9) | −0.0024 (7) | 0.0086 (7) | 0.0152 (7) |
| O3 | 0.0334 (9) | 0.0306 (9) | 0.0232 (8) | 0.0000 (7) | 0.0118 (7) | 0.0019 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O4 | 0.0383 (10) | 0.0439 (11) | 0.0231 (8) | −0.0078 (8) | 0.0124 (7) | −0.0015 (7) |
| O5 | 0.0724 (14) | 0.0362 (10) | 0.0262 (9) | −0.0092 (10) | 0.0121 (9) | −0.0138 (8) |
| O6 | 0.0376 (10) | 0.0214 (8) | 0.0315 (9) | −0.0086 (7) | 0.0036 (7) | −0.0039 (7) |
| N1 | 0.0232 (10) | 0.0171 (9) | 0.0219 (9) | −0.0036 (7) | 0.0039 (8) | 0.0027 (7) |
| N2 | 0.0260 (10) | 0.0176 (9) | 0.0226 (9) | −0.0041 (8) | 0.0069 (8) | 0.0029 (7) |
| N3 | 0.0166 (9) | 0.0137 (8) | 0.0181 (9) | −0.0016 (7) | 0.0019 (7) | 0.0022 (7) |
| N4 | 0.0177 (9) | 0.0148 (9) | 0.0194 (9) | −0.0006 (7) | 0.0010 (7) | 0.0013 (7) |
| C1 | 0.0142 (10) | 0.0191 (10) | 0.0197 (10) | 0.0005 (8) | 0.0003 (8) | 0.0049 (8) |
| C2 | 0.0172 (11) | 0.0219 (11) | 0.0203 (11) | −0.0007 (9) | 0.0046 (9) | 0.0018 (9) |
| C3 | 0.0175 (11) | 0.0172 (10) | 0.0270 (11) | −0.0017 (8) | 0.0006 (9) | 0.0044 (9) |
| C4 | 0.0152 (10) | 0.0232 (11) | 0.0256 (11) | −0.0002 (8) | 0.0031 (9) | 0.0086 (9) |
| C5 | 0.0187 (11) | 0.0252 (12) | 0.0181 (10) | 0.0043 (9) | 0.0023 (9) | 0.0049 (9) |
| C6 | 0.0206 (11) | 0.0181 (10) | 0.0193 (11) | 0.0014 (9) | −0.0008 (9) | 0.0025 (8) |
| C7 | 0.0317 (13) | 0.0234 (12) | 0.0373 (14) | −0.0052 (10) | 0.0068 (11) | −0.0041 (10) |
| C8 | 0.0365 (14) | 0.0379 (14) | 0.0351 (14) | −0.0037 (11) | 0.0067 (11) | 0.0187 (11) |
| C9 | 0.0519 (17) | 0.0324 (14) | 0.0218 (12) | 0.0057 (12) | 0.0101 (11) | −0.0018 (10) |
| C10 | 0.0184 (11) | 0.0192 (10) | 0.0177 (10) | 0.0007 (8) | 0.0004 (8) | 0.0018 (8) |
| C11 | 0.0174 (10) | 0.0161 (10) | 0.0205 (10) | −0.0034 (8) | 0.0005 (8) | 0.0060 (8) |
| C12 | 0.0159 (10) | 0.0175 (10) | 0.0195 (10) | 0.0008 (8) | −0.0010 (8) | 0.0049 (8) |
| C13 | 0.0155 (10) | 0.0203 (11) | 0.0181 (10) | 0.0007 (8) | −0.0017 (8) | 0.0028 (8) |
| C14 | 0.0189 (11) | 0.0260 (11) | 0.0207 (11) | −0.0031 (9) | 0.0001 (9) | 0.0034 (9) |
| C15 | 0.0228 (12) | 0.0371 (13) | 0.0179 (11) | −0.0019 (10) | 0.0032 (9) | 0.0006 (9) |
| C16 | 0.0325 (13) | 0.0310 (13) | 0.0204 (11) | −0.0034 (10) | 0.0021 (10) | −0.0072 (10) |
| C17 | 0.0249 (12) | 0.0190 (11) | 0.0253 (12) | −0.0028 (9) | −0.0031 (9) | −0.0020 (9) |
| C18 | 0.0181 (11) | 0.0227 (11) | 0.0198 (10) | 0.0006 (9) | 0.0003 (9) | 0.0025 (9) |
| C19 | 0.0391 (10) | 0.0433 (10) | 0.0349 (9) | −0.0054 (8) | 0.0068 (8) | 0.0050 (8) |
| C20 | 0.0504 (11) | 0.0500 (11) | 0.0512 (11) | 0.0040 (9) | 0.0021 (9) | −0.0090 (9) |
| C21 | 0.0274 (13) | 0.0211 (11) | 0.0357 (13) | −0.0031 (10) | −0.0010 (10) | 0.0035 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C11 | 1.729 (2) | C6—H6 | 0.9300 |
| S1—C12 | 1.766 (2) | C7—H7A | 0.9600 |
| O1—C3 | 1.372 (3) | C7—H7B | 0.9600 |
| O1—C7 | 1.426 (3) | C7—H7C | 0.9600 |
| O2—C4 | 1.374 (3) | C8—H8A | 0.9600 |
| O2—C8 | 1.422 (3) | C8—H8B | 0.9600 |
| O3—C5 | 1.369 (3) | C8—H8C | 0.9600 |
| O3—C9 | 1.427 (3) | C9—H9A | 0.9600 |
| O4—C15 | 1.369 (3) | C9—H9B | 0.9600 |
| O4—C19 | 1.420 (3) | C9—H9C | 0.9600 |
| O5—C16 | 1.372 (3) | C12—C13 | 1.465 (3) |
| O5—C20 | 1.456 (4) | C13—C18 | 1.394 (3) |
| O6—C17 | 1.371 (3) | C13—C14 | 1.395 (3) |
| O6—C21 | 1.426 (3) | C14—C15 | 1.384 (3) |
| N1—C10 | 1.319 (3) | C14—H14 | 0.9300 |
| N1—N2 | 1.394 (3) | C15—C16 | 1.394 (3) |
| N2—C11 | 1.313 (3) | C16—C17 | 1.395 (3) |

| | | | |
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| N3—C11 | 1.363 (3) | C17—C18 | 1.382 (3) |
| N3—N4 | 1.374 (2) | C18—H18 | 0.9300 |
| N3—C10 | 1.379 (3) | C19—H19A | 0.9600 |
| N4—C12 | 1.299 (3) | C19—H19B | 0.9600 |
| C1—C2 | 1.389 (3) | C19—H19C | 0.9600 |
| C1—C6 | 1.400 (3) | C20—H20A | 0.9600 |
| C1—C10 | 1.460 (3) | C20—H20B | 0.9600 |
| C2—C3 | 1.387 (3) | C20—H20C | 0.9600 |
| C2—H2 | 0.9300 | C21—H21A | 0.9600 |
| C3—C4 | 1.393 (3) | C21—H21B | 0.9600 |
| C4—C5 | 1.403 (3) | C21—H21C | 0.9600 |
| C5—C6 | 1.380 (3) | | |

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| C11—S1—C12 | 87.61 (10) | H9A—C9—H9C | 109.5 |
| C3—O1—C7 | 116.73 (17) | H9B—C9—H9C | 109.5 |
| C4—O2—C8 | 114.35 (17) | N1—C10—N3 | 107.57 (18) |
| C5—O3—C9 | 116.88 (18) | N1—C10—C1 | 127.0 (2) |
| C15—O4—C19 | 116.8 (2) | N3—C10—C1 | 125.41 (19) |
| C16—O5—C20 | 115.3 (2) | N2—C11—N3 | 110.97 (18) |
| C17—O6—C21 | 116.75 (17) | N2—C11—S1 | 139.70 (16) |
| C10—N1—N2 | 109.70 (17) | N3—C11—S1 | 109.32 (15) |
| C11—N2—N1 | 105.61 (16) | N4—C12—C13 | 121.29 (19) |
| C11—N3—N4 | 118.40 (17) | N4—C12—S1 | 116.85 (16) |
| C11—N3—C10 | 106.15 (17) | C13—C12—S1 | 121.85 (16) |
| N4—N3—C10 | 135.43 (17) | C18—C13—C14 | 120.9 (2) |
| C12—N4—N3 | 107.81 (17) | C18—C13—C12 | 118.23 (19) |
| C2—C1—C6 | 120.74 (19) | C14—C13—C12 | 120.9 (2) |
| C2—C1—C10 | 121.14 (19) | C15—C14—C13 | 119.4 (2) |
| C6—C1—C10 | 118.12 (19) | C15—C14—H14 | 120.3 |
| C3—C2—C1 | 119.3 (2) | C13—C14—H14 | 120.3 |
| C3—C2—H2 | 120.4 | O4—C15—C14 | 124.2 (2) |
| C1—C2—H2 | 120.4 | O4—C15—C16 | 115.5 (2) |
| O1—C3—C2 | 123.7 (2) | C14—C15—C16 | 120.3 (2) |
| O1—C3—C4 | 115.37 (19) | O5—C16—C15 | 119.0 (2) |
| C2—C3—C4 | 120.9 (2) | O5—C16—C17 | 121.3 (2) |
| O2—C4—C3 | 120.4 (2) | C15—C16—C17 | 119.6 (2) |
| O2—C4—C5 | 120.5 (2) | O6—C17—C18 | 124.0 (2) |
| C3—C4—C5 | 118.98 (19) | O6—C17—C16 | 115.1 (2) |
| O3—C5—C6 | 124.52 (19) | C18—C17—C16 | 120.8 (2) |
| O3—C5—C4 | 114.81 (19) | C17—C18—C13 | 119.0 (2) |
| C6—C5—C4 | 120.67 (19) | C17—C18—H18 | 120.5 |
| C5—C6—C1 | 119.37 (19) | C13—C18—H18 | 120.5 |
| C5—C6—H6 | 120.3 | O4—C19—H19A | 109.5 |
| C1—C6—H6 | 120.3 | O4—C19—H19B | 109.5 |
| O1—C7—H7A | 109.5 | H19A—C19—H19B | 109.5 |
| O1—C7—H7B | 109.5 | O4—C19—H19C | 109.5 |
| H7A—C7—H7B | 109.5 | H19A—C19—H19C | 109.5 |
| O1—C7—H7C | 109.5 | H19B—C19—H19C | 109.5 |

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| H7A—C7—H7C | 109.5 | O5—C20—H20A | 109.5 |
| H7B—C7—H7C | 109.5 | O5—C20—H20B | 109.5 |
| O2—C8—H8A | 109.5 | H20A—C20—H20B | 109.5 |
| O2—C8—H8B | 109.5 | O5—C20—H20C | 109.5 |
| H8A—C8—H8B | 109.5 | H20A—C20—H20C | 109.5 |
| O2—C8—H8C | 109.5 | H20B—C20—H20C | 109.5 |
| H8A—C8—H8C | 109.5 | O6—C21—H21A | 109.5 |
| H8B—C8—H8C | 109.5 | O6—C21—H21B | 109.5 |
| O3—C9—H9A | 109.5 | H21A—C21—H21B | 109.5 |
| O3—C9—H9B | 109.5 | O6—C21—H21C | 109.5 |
| H9A—C9—H9B | 109.5 | H21A—C21—H21C | 109.5 |
| O3—C9—H9C | 109.5 | H21B—C21—H21C | 109.5 |

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| C10—N1—N2—C11 | 0.1 (2) | N4—N3—C11—N2 | 177.96 (17) |
| C11—N3—N4—C12 | 0.1 (2) | C10—N3—C11—N2 | −0.6 (2) |
| C10—N3—N4—C12 | 178.2 (2) | N4—N3—C11—S1 | −0.9 (2) |
| C6—C1—C2—C3 | 0.8 (3) | C10—N3—C11—S1 | −179.49 (13) |
| C10—C1—C2—C3 | −178.50 (19) | C12—S1—C11—N2 | −177.3 (3) |
| C7—O1—C3—C2 | 10.3 (3) | C12—S1—C11—N3 | 1.03 (15) |
| C7—O1—C3—C4 | −171.13 (19) | N3—N4—C12—C13 | −179.48 (17) |
| C1—C2—C3—O1 | 178.88 (19) | N3—N4—C12—S1 | 0.8 (2) |
| C1—C2—C3—C4 | 0.4 (3) | C11—S1—C12—N4 | −1.12 (17) |
| C8—O2—C4—C3 | 86.6 (3) | C11—S1—C12—C13 | 179.16 (18) |
| C8—O2—C4—C5 | −97.3 (2) | N4—C12—C13—C18 | −2.4 (3) |
| O1—C3—C4—O2 | −4.2 (3) | S1—C12—C13—C18 | 177.34 (15) |
| C2—C3—C4—O2 | 174.44 (19) | N4—C12—C13—C14 | 176.02 (19) |
| O1—C3—C4—C5 | 179.64 (19) | S1—C12—C13—C14 | −4.3 (3) |
| C2—C3—C4—C5 | −1.7 (3) | C18—C13—C14—C15 | −1.1 (3) |
| C9—O3—C5—C6 | 3.6 (3) | C12—C13—C14—C15 | −179.42 (19) |
| C9—O3—C5—C4 | −175.7 (2) | C19—O4—C15—C14 | −8.6 (3) |
| O2—C4—C5—O3 | 5.2 (3) | C19—O4—C15—C16 | 171.6 (2) |
| C3—C4—C5—O3 | −178.67 (18) | C13—C14—C15—O4 | 179.6 (2) |
| O2—C4—C5—C6 | −174.19 (18) | C13—C14—C15—C16 | −0.6 (3) |
| C3—C4—C5—C6 | 2.0 (3) | C20—O5—C16—C15 | 119.7 (3) |
| O3—C5—C6—C1 | 179.86 (19) | C20—O5—C16—C17 | −63.8 (3) |
| C4—C5—C6—C1 | −0.9 (3) | O4—C15—C16—O5 | −1.3 (3) |
| C2—C1—C6—C5 | −0.5 (3) | C14—C15—C16—O5 | 178.9 (2) |
| C10—C1—C6—C5 | 178.77 (18) | O4—C15—C16—C17 | −177.9 (2) |
| N2—N1—C10—N3 | −0.4 (2) | C14—C15—C16—C17 | 2.3 (4) |
| N2—N1—C10—C1 | −179.76 (19) | C21—O6—C17—C18 | −1.0 (3) |
| C11—N3—C10—N1 | 0.6 (2) | C21—O6—C17—C16 | −179.9 (2) |
| N4—N3—C10—N1 | −177.6 (2) | O5—C16—C17—O6 | 0.1 (3) |
| C11—N3—C10—C1 | 179.98 (19) | C15—C16—C17—O6 | 176.6 (2) |
| N4—N3—C10—C1 | 1.7 (4) | O5—C16—C17—C18 | −178.8 (2) |
| C2—C1—C10—N1 | 176.3 (2) | C15—C16—C17—C18 | −2.3 (4) |
| C6—C1—C10—N1 | −3.1 (3) | O6—C17—C18—C13 | −178.1 (2) |
| C2—C1—C10—N3 | −2.9 (3) | C16—C17—C18—C13 | 0.7 (3) |
| C6—C1—C10—N3 | 177.75 (18) | C14—C13—C18—C17 | 1.1 (3) |

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|---|---|---|---|
| N1—N2—C11—N3 | 0.4 (2) | C12—C13—C18—C17 | 179.43 (19) |
| N1—N2—C11—S1 | 178.7 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2···N4 | 0.93 | 2.36 | 3.047 (3) | 130 |
| C14—H14···S1 | 0.93 | 2.72 | 3.130 (3) | 108 |
| C19—H19*B*···O3$^{i}$ | 0.96 | 2.53 | 3.384 (2) | 148 |
| C21—H21*B*···O1$^{ii}$ | 0.96 | 2.43 | 3.331 (3) | 156 |
| C19—H19*C*···*Cg*3$^{iii}$ | 0.96 | 3.30 | 4.057 (3) | 137 |

Symmetry codes: (i) *x*+1, *y*, *z*−1; (ii) −*x*, −*y*, −*z*+2; (iii) −*x*+1, −*y*+1, −*z*.